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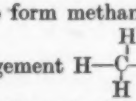
MODERN METHODS OF MOTOR FUEL PRODUCTION.

By Gustav Egloff (Fellow).

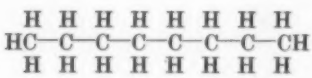
Hydrocarbons in Petroleum.

Crude petroleums, the materials that are the basis for such a variety of commercial products as motor fuels, kerosines, furnace oils, lubricants, waxes, fuel oils, and asphalts, are mixtures in various proportions of different gaseous, liquid, and solid hydrocarbons, each of which has its own density, boiling point, melting point, fluidity, combustion characteristics, and stability when heated. As the name *hydrocarbon* suggests, these various compounds consist of the element carbon, as represented by diamond, graphite, and coke, and the element hydrogen, which is the lightest known gas. In a compound, which is a substance characterized by definite values in regard to density, boiling point, melting point, viscosity (if a liquid), and crystal form (if a solid), each molecule, representing the smallest mechanically subdivisible particle of the material, is composed of a fixed number of atoms in a particular pattern or structural arrangement. To understand petroleum processes properly, one should have some knowledge of the various types of hydrocarbons, as well as information as to which types are found in petroleum oils and their refined products, and which types impart the most desirable properties to motor fuels, lubricants, and other petroleum products.

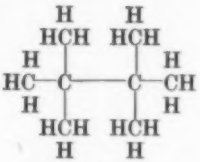
To form hydrocarbons, the element carbon, having the chemical symbol "C," and the element hydrogen, with the symbol "H," combine by virtue of their valences, or combining strengths, the valence of the hydrogen atom being taken as one. Since a carbon atom is able to combine with four hydrogen atoms to form methane, having the formula CH_4 and the presumed atomic arrangement  , it has a valence of four. This compound, known as methane, is the principal constituent of natural gas. Some of the carbon atoms in hydrocarbons combine with hydrogen, and some with each other. Because carbon atoms can combine in this manner, it is possible to form a large number of molecules of different size and structure. In fact, almost every arrangement that can be indicated on paper represents a possible compound.

Molecules of hydrocarbons are said to be *saturated* when the carbon atoms are combined by just one of their four valences, and their remaining valences are absorbed by hydrogen atoms. When the carbon atoms in a saturated hydrocarbon are in rows, they form paraffins or alkanes. Some of these compounds have an extended structure, and others have a

more compact arrangement, as indicated in the two formulæ given below:



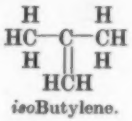
Normal Octane.
Extended-chain structure.



An *iso*-Octane.
Compact or branched-chain structure.

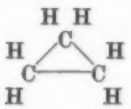
In some compounds there is not sufficient hydrogen to satisfy all valences of the carbon atoms, and adjoining carbon atoms exert more than one valence on each other. Such compounds are said to be *unsaturated* with respect to hydrogen, and are designated as olefins or alkenes. Typical olefin structures are shown below:

Ethylene.

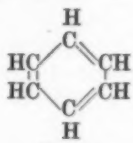


*iso*Butylene.

The compacting of carbon atoms may result in the formation of molecules having the so-called ring structure; these compounds are known as *cyclo*-paraffins and aromatics as represented in the formulæ given below:



*cyclo*Propane
(a *cyclo*paraffin).



Benzene
(an aromatic).

The properties of a hydrocarbon depend on the number of carbon atoms its molecules contain, the ratio of carbon to hydrogen atoms, and their positions with respect to each other. In general, as the carbon atoms increase in number, the relative density, boiling point, and melting point increase. This rule, however, applies only to compounds having similar structures. The compacting of a given number of hydrogen and carbon atoms influences the density and also the boiling point. The hydrogen deficiency in unsaturated hydrocarbons also affects their properties, sometimes in one direction, sometimes in another.

Refining of Petroleum.

The primary step in refining a crude petroleum involves its separation into portions, or *fractions*, having different boiling-point ranges. The crude oil is pumped through a coil of pipe disposed in a furnace, and the temperature of the oil is raised to about 500–700° F. The heated oil is

then introduced into a *fractionating column*; and therein hydrocarbons having different boiling points separate or stratify so that the lower-boiling gaseous and gasoline hydrocarbons are recovered from the upper part of the column, while progressively higher-boiling fractions, such as kerosine, furnace oils, and lubricants, are recovered from lower exits, heavy liquid fuel oils or asphalts being drawn off as a residue. By this initial procedure a rough separation of groups of hydrocarbons is accomplished, and the properties of the different groups are the sum of the properties of the individual compounds present. Fig. 1 shows the general flow of charge and products in a primary fractionation of crude petroleum. The gasoline boiling-range fractions produced in the primary distillation of the great majority of crude petroleums are not suitable for use in high-compression automobile and airplane engines. These light fractions,

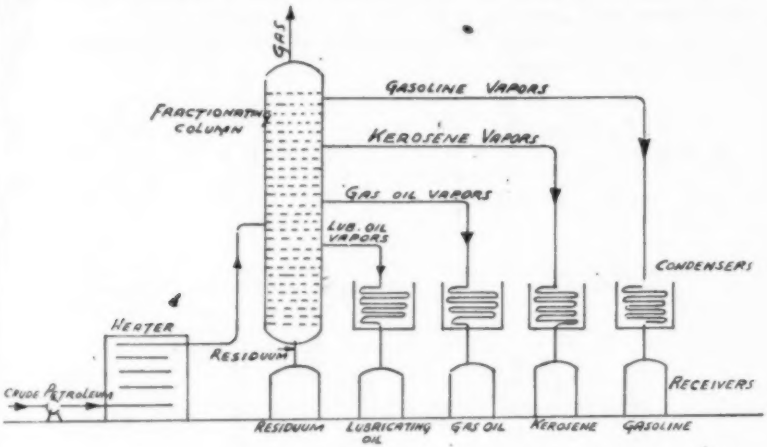


FIG. 1.

PRIMARY FRACTIONATION OF CRUDE PETROLEUM.

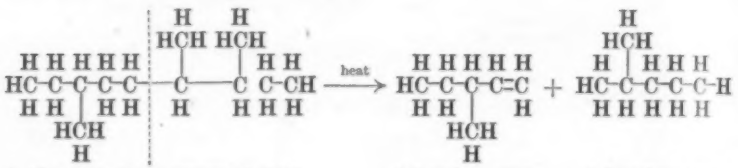
boiling from about 100° to 400° F., are composed for the most part of saturated hydrocarbons of open and extended structures; and such compounds are notoriously bad "knockers" in high-compression engines. Engine tests with hydrocarbons of known structure have shown that, in general, the better types from an anti-knock standpoint are those having either an unsaturated character or a more compact molecular structure, or both. Such hydrocarbons, if present at all in the primary, or "straight-run," gasolines, are there in only minor amounts, so that knocking tendencies are principally determined by the hydrocarbons of a straight-chain saturated character.

Cracking.

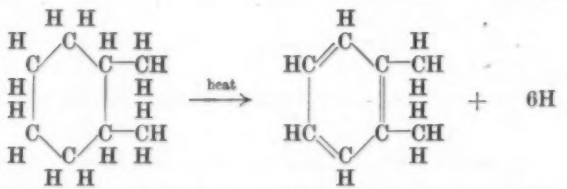
The major object in petroleum refining at the present time is the production of maximum yields of gasoline having high anti-knock properties, and lubricants for use in various types of machinery. The basic process

utilized for increasing the yield of gasoline is the cracking process, in which the higher-boiling fractions from the primary crude petroleum distillation are heated to high temperatures to decompose large hydrocarbon molecules into smaller ones. As the carbon atoms in hydrocarbons increase in number, the molecules become more unwieldy and less stable; so that when they are heated at elevated temperatures and pressures for a time, they break down into smaller and more stable molecules having lower boiling points and constituting cracked gasoline. In the breaking-up of heavier molecules, hydrogen atoms are split off, carbon atom combinations are disrupted, and transient carbon–hydrogen groupings are recombined to form compounds having different properties. The net result is the production of both low-boiling unsaturated chain compounds and ring compounds, so that the anti-knock value of a cracked gasoline is practically always higher than that of the primary, or straight-run, gasoline produced from the original crude oil in its primary fractionation.

Some typical break-ups of hydrocarbons in cracking are represented in the following equations:



A 12-carbon-atom saturated chain hydrocarbon. | A 6-carbon-atom unsaturated chain hydrocarbon. | A 6-carbon-atom saturated chain hydrocarbon.



A 6-carbon-atom saturated ring hydrocarbon. | A 6-carbon-atom unsaturated ring hydrocarbon. | Hydrogen.

The first reaction involves a break between carbon atoms with a formation of two lower-boiling molecules, one saturated and the other unsaturated, while the second equation represents a typical dehydrogenation reaction. The net result of the two reactions is an improvement in the anti-knock values of the products, both because the compounds formed have lower boiling points and because they are unsaturated.

While the above two types of reactions are typical of those occurring during the cracking of hydrocarbons, other reactions of a more complicated character occur, corresponding in some instances to the complete degradation of the heavier molecules and separation of highly carbonaceous solids having the characteristics of coke. In the present discussion, emphasis is being placed on the rôle of cracking and auxiliary processes in producing

hydrocarbons of higher anti-knock value, due to their more condensed structures. However, suitable motor fuels must also possess a definite volatility as shown by their boiling-point ranges, and must also have only small amounts of gum-forming components and sulphur. A detailed discussion of the total properties required in commercial motor fuels would be outside the scope of this paper.

Fig. 2 shows the flows in a typical commercial thermal cracking unit.[1] A topped crude (a residuum from a primary petroleum distillation) is introduced directly into a fractionating column, where it undergoes fractionation together with the cracked products. Cracked gasoline and a furnace oil are recovered from the upper portion of the column, while light- and heavy-oil fractions are withdrawn from the lower levels and pumped to separate heaters, wherein they are subjected to temperatures best adapted to their cracking. The combined products from the two heaters

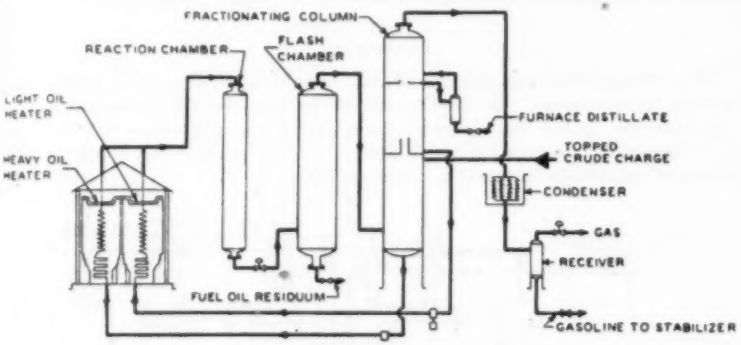


FIG. 2.

FLOW DIAGRAM OF TWO-COIL SELECTIVE DUBBS CRACKING UNIT.

are passed to a reaction chamber, in which time is given for the cracking reactions to progress to an optimum point. The total cracked products are released into a flash-chamber at lower pressure, from which residual fuel oil is drawn, and the remaining vaporous products are sent to the fractionating column for further distillation. The gas mixture vented from the gasoline receiver contains many of the compounds used in synthetic processes, to be described later. These gases are essentially the lighter hydrocarbon fragments produced by the breaking down of large hydrocarbon molecules, and contain hydrogen and both paraffins and olefins.

As a result of the cracking of the higher-boiling fractions of petroleum not needed for other uses, the yield of gasoline, including both the straight-run and the cracked varieties, may be as high as 50 or 60 per cent. by volume of the original crude oil. Since the average crude oil contains about 25–30 per cent. of gasoline boiling up to 400° F., it is evident that the thermal cracking process doubles the yield of gasoline. Temperatures varying from about 850° to about 1000° F., and pressures varying from about 100 lb. to about 500 lb. per square inch, are used in most of these processes.

The applicability of the thermal cracking process to the production of gasoline is limited by the character of the hydrocarbons cracked, particularly their carbon–hydrogen ratios. By the best application of thermal cracking, a yield of 60–65 per cent. by volume of 70–75 octane-number motor fuel can be obtained. If attempts are made to increase the octane number by using more severe cracking conditions, such as higher temperatures, higher pressures, and longer times in processing, the yield of gasoline drops off. When less severe cracking conditions are used, higher yields may be obtained, but with correspondingly lower octane numbers.

The anti-knock values, or *octane numbers*, of gasolines are determined by comparing their knocking tendencies with mixtures of normal heptane, a knocking paraffin hydrocarbon which is given a zero rating, and *iso*-octane, a non-knocking paraffin hydrocarbon which is given a rating of 100. The percentage of *iso*-octane in the blend that knocks to the same extent as the gasoline under test is taken as its octane number.

Reforming.

If a straight-run gasoline is suitably heat-treated, a process commonly known as *reforming*, the anti-knock value may be increased, although considerable losses are suffered because of gas formation. However, with intensified demands for high anti-knock value material, it may sometimes be profitable to take the losses in yield.

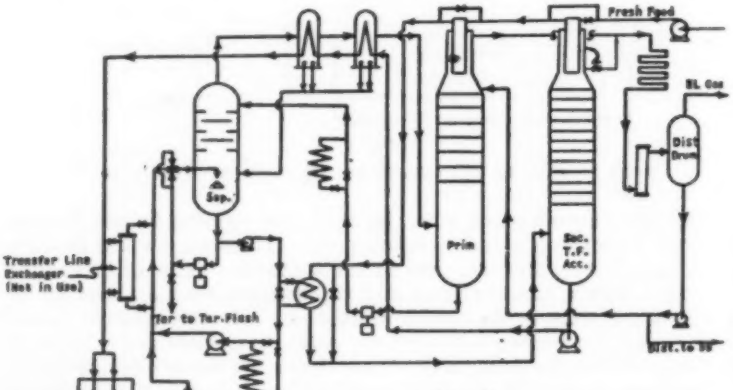


FIG. 3.

REFORMING.

Fig. 3 shows the flow in a commercial plant which is used for reforming a 300° F. end boiling-point gasoline to obtain very high anti-knock-value material for blending in aviation fuels.[2] The gasoline is preheated by passing it through heat exchangers positioned in the upper portions of successive fractionating columns. It is then introduced directly into the second column, wherein it undergoes fractionation with the products of

the reforming process. The residual portions from the second column are then further preheated by heat exchange with the vapours from a tar separator, and the preheated charge is then divided and passed in parallel flow through a two-sectional heater, in which the temperature is raised to 1100° F. and the pressure maintained at 400 lb. per square inch. A plant of this character ran continuously for six months, during which time the octane number of a million 42-gallon barrels of gasoline was raised from 62 to 92. At the same time 90,000 barrels of liquid butylenes (four-carbon-atom olefin hydrocarbons) were produced, which were further processed to increase the gasoline yield.

The gases produced in the cracking and reforming process contain varying proportions of hydrogen, methane, and paraffin and olefin hydrocarbons containing two-, three-, and four-carbon atoms per molecule.

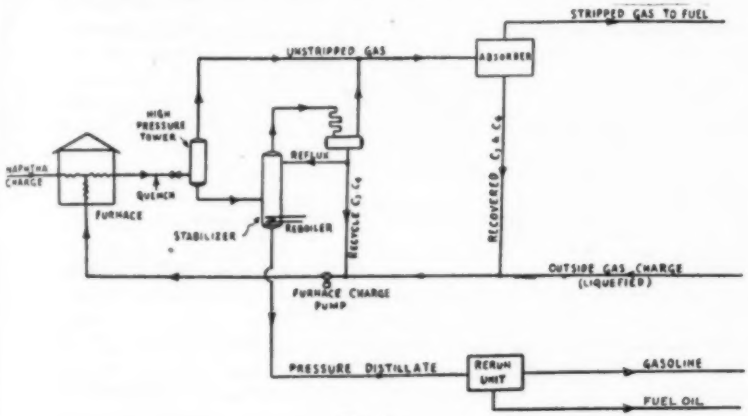


FIG. 4.

SCHEMATIC FLOW OF NAPHTHA–GAS REVERSION UNIT.

The gaseous paraffin hydrocarbons containing two-, three-, and four-carbon atoms per molecule include ethane (C_2H_6), propane (C_3H_8), and butanes (C_4H_{10}); and the gaseous olefins include ethylene (C_2H_4), propylene (C_3H_6), and butylenes (C_4H_8). Large amounts of the paraffins are present in natural gases, petroleum-distillation gases, and cracking-process gases; and the last named contain important quantities of the olefins listed. When these gases are converted into liquid products, the yields of motor fuel from crude oils are considerably augmented. It has been found that when they are heated at high temperatures (above 1000° F.) and high pressures (300–500 lb. per square inch), they undergo decompositions and recompositions similar to those occurring in oil cracking, although the yields of liquid products are relatively low. An effective method for obtaining gasoline from hydrocarbon gases is to crack them together with naphthas which are being reformed to improve their anti-knock ratings.

Fig. 4 shows the flow in a plant designed to reform naphtha (in this case a gasoline fraction) and, at the same time, convert a mixture of hydro-

carbon gases consisting principally of three- and four-carbon-atom paraffins into liquid hydrocarbons (gas-reversion).[4] The naphtha is partly cracked in passing through the primary heater section. The separately heated hydrocarbon gas is then introduced into the stream of naphtha, and the mixture is finally heated at an increased temperature to complete the naphtha-reforming and gas-cracking reactions. After a relatively cool quench oil is introduced to arrest the conversion reactions, the products are fractionated in distilling and absorbing equipment to recover gasoline, and undecomposed three- and four-carbon-atom hydrocarbons are recycled for further conversion treatment.

Fig. 5 shows a modified flow for simultaneous naphtha reforming and gas conversion in which the naphtha charge is first used as an absorbing

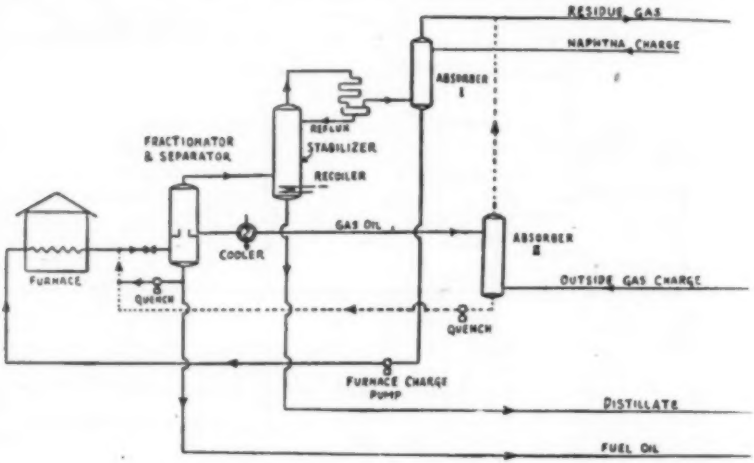


FIG. 5.

SCHEMATIC FLOW OF NAPHTHA POLYFORM UNIT.

oil to take up the more soluble three- and four-carbon-atom hydrocarbons in the process gases and carry them in solution to the heating unit, while a cooled intermediate gas-oil fraction is used to absorb similar gases from an outside gas charge and then used as a quenching medium.[4] The more soluble three- and four-carbon-atom hydrocarbons are thus introduced to the plant, and later converted together with the naphtha. The desired gasoline product is recovered from the bottom of the stabilizer, from the top of which gases are passed to the primary naphtha absorber. From this final step light gases are vented as residue gas, since they are of little value in the production of gasoline.

Catalytic Cracking.

The processes thus far described are of a purely thermal character, depending on the use of temperature, pressure, and time in suitable re-

lationships for their successful operation. At the present time increasing use is being made of *catalysts* to accelerate various hydrocarbon conversion reactions in the direction of increased yields and improved qualities of products. The word catalyst comes from a Greek verb meaning literally to "loosen up," and refers to the fact that small amounts of certain materials introduced into a reaction zone markedly speed up the reactions. Catalytic materials used in hydrocarbon processes are usually innocent-looking powders or granules which exemplify the old adage "a little bit goes a long way"; for when they are present in relatively small amounts in a conversion process, the conversions take place more rapidly, and temperature, pressure, and time can all be reduced to get the same effects. When the reactions are completed, the catalyst has usually undergone no perceptible change; and how it performs its function is still a matter of dispute among scientists.

Much experimenting was done in attempts to find catalysts that would increase the rate of the more desirable reactions occurring in cracking before suitable materials were found, since there are practically no rules for predicting what type of catalyst will accelerate a given reaction. Metals, oxides, sulphides, salts, ores, and earths were all tried with varying degrees of success. At the present time the best materials appear to be certain types of clays and manufactured aluminium silicates or composites of silica and alumina. Such materials can be made porous and absorbent; they are refractory at the high temperatures employed in cracking processes; and they function for a long time without too great loss in activity. Furthermore, they "loosen up" the cracking reactions so that types of low-boiling molecules are formed that are different from those formed in non-catalytic cracking; and it has been found that higher octane-number cracked gasolines are obtainable.

Catalytic cracking processes in a sense supplement the strictly thermal processes employing no catalysts. The cracking processes, both thermal and thermal-catalytic, are still the principal processes used for increasing the yield of gasoline from petroleum. The development of these processes in their present degree of commercial efficiency has been due to the combined efforts of the research chemists and engineers of many organizations, such as the Universal Oil Products Co., and the large oil refiners.

The catalyst may be used in different ways to accelerate and give direction to cracking reactions. In the earliest-devised and perhaps the simplest method of operation, the catalyst in the form of small granules or pills is placed in the reaction chamber following the tubular heating element, and the preheated oil vapours are passed through the stationary bed of catalyst particles, usually in a downward direction. Carbon and tarry substances are slowly deposited on the catalyst particles as cracking proceeds; and as their surfaces gradually become coated with these materials, they become less active and the reactions revert to those of a strictly thermal character. When such a point is reached, the oil vapours are diverted into an auxiliary chamber containing clean catalyst, and the carbon-coated particles in the other chamber are contacted with heated air to burn off carbonaceous material from their surfaces and restore their activity for further use.

This method is illustrated in Fig. 6, which shows the essential flow in

such a "fixed-bed" catalytic cracking plant.[5] A gas-oil charge is preheated by exchange with the cracked products from the catalyst towers, is vaporized, and is heated to catalytic cracking temperature during passage through a pipe heater; then it is passed alternately through one or the other of two towers containing granular catalyst particles to effect the production of high-anti-knock-value gasoline. A fractionating column and a stabilizer for removing any excess of dissolved gaseous hydrocarbons complete the plant.

In an improved commercial modification of the catalytic cracking process, known as the "fluid process," the catalyst is employed in the form of a very fine powder, which is carried upward in a stream of heated oil vapours during their passage through a reactor in which cracking takes place. The carbonized powder is separated by settling in an enlarged

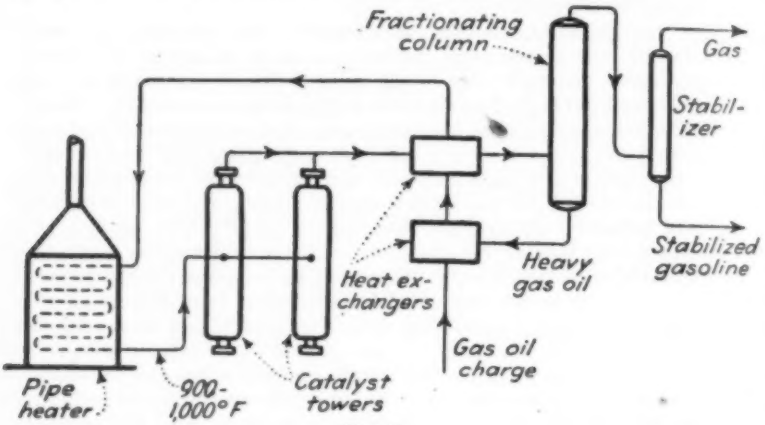


FIG. 6.

CATALYTIC CRACKING OF GAS-OIL STOCKS BY THE STATIC BED PROCESS.

chamber, assisted by centrifugal or electrical influences, and the cracked products are fractionated to recover gasoline and other cracked products. For the decarbonization of the separated catalyst powder, it is carried upward in a stream of heated air through a regeneration zone, and then separated and re-used for further cracking. This process can be operated continuously without shifting the stream of oil vapours from one reactor to another as in the fixed-bed operation.

Fig. 7 shows the essential flow of a process of this character, with the exception of the fractionating equipment.[3] In still another modification of the catalytic cracking process, a mass of granular catalyst particles moves downward through the cracking zone, counterflow to a rising stream of heated oil vapours. The carbonized particles are separated and elevated by mechanical conveyers, after which they move downward countercurrent to a rising stream of heated air in a regenerator. They are then separated from the combustion gases and conveyed back to the top of the reactor to complete their cycle and make possible continuous operation.

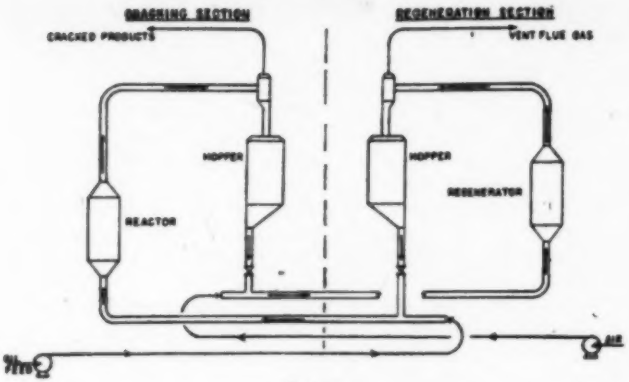


FIG. 7.

CATALYST FLOW IN FLUID CRACKING PROCESS.

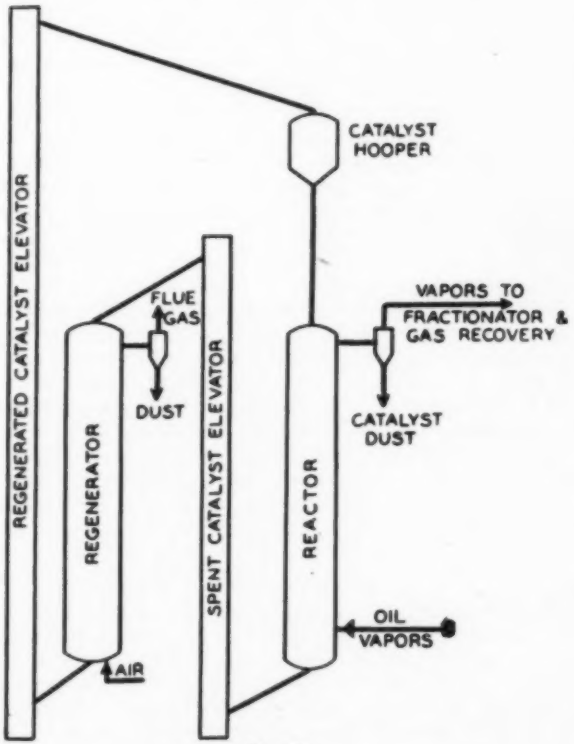


FIG. 8.

MOVING BED CATALYTIC CRACKING PROCESS.

The essential flows of catalyst, oil vapours, and regenerating gases are shown in Fig. 8.[7]

The chief advantage enjoyed by catalytic cracking processes is that gasoline can be obtained having much higher anti-knock values than those produced in strictly thermal cracking. In cracking without catalysts the upper limit of octane number is about 75, whereas the use of catalysts permits the production of gasolines having octane numbers as high as 90, this being a decided improvement, since the relative utility of the 90-octane material is much greater than the ratio of the two values. Besides producing better grades of gasoline, the catalytic cracking process yields gaseous products that are more easily converted into further yields of high-octane material.

Cracking processes, particularly those employing catalysts to modify the character of the motor-fuel fractions produced, have now been supplemented by auxiliary catalytic conversion processes, which either utilize the gaseous cracked products in the production of more gasoline, or convert primary or cracked gasoline fractions into higher anti-knock value materials. Some of these processes will now be reviewed.

Polymerization.

One of the earliest ways of utilizing cracking process gases for the production of more gasoline was the catalytic *polymerization* process, in which gaseous olefins are combined to make gasoline boiling-range liquids. The word polymer is from the Greek, and means literally "many times." The polymerization process, therefore, involves the combination of several molecules in definite multiple proportions to make larger ones of similar character having higher boiling points. Thus, through the use of certain acids, such as sulphuric or phosphoric acid, as catalysts, two molecules of a butylene, which is a four-carbon-atom gaseous olefin, may be combined to form eight-carbon-atom liquid olefins boiling above 200° F.

Fig. 9 illustrates the essential flow in a catalytic polymerization plant employing a solid granular phosphoric acid catalyst for the polymerization of three- and four-carbon-atom olefins in a cracked-gas mixture to produce additional yields of gasoline.[5] A cracked gasoline containing such olefins in solution is first mildly heated, and olefin-containing gases are distilled out of the stabilizer. The gases are then compressed and forced through the pipe-heater, after which they are contacted with the catalyst in a series of catalyst towers to produce liquid olefins. The liquid products, after being cooled, pass through the stabilizer to remove any excess of gases over that necessary for assuring a proper vapour pressure.

Dehydrogenation.

The *dehydrogenation* process was developed for making additional yields of polymerizable olefins from the corresponding gaseous paraffins present in hydrocarbon gas mixtures in order to produce further yields of gasoline. In Fig. 10 the flow of such a process is shown.[6] Butanes separated from natural, refinery, or cracking-plant gases are heated to temperatures of the order of 1000–1100° F., depending on their composition; they then pass through reactors containing a granular chromium oxide catalyst,

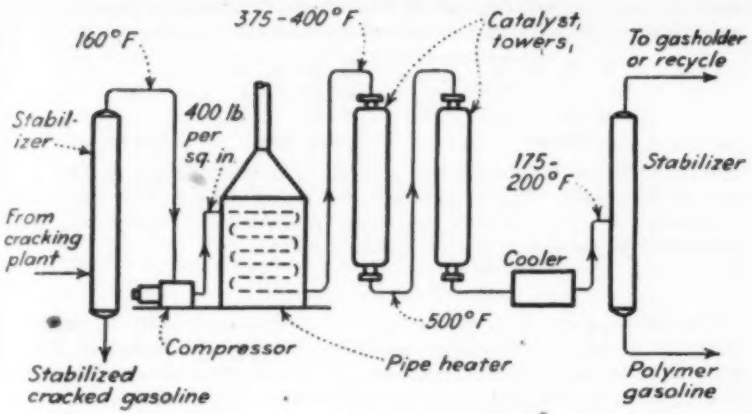


FIG. 9.

FLOW SHEET FOR CATALYTIC POLYMERIZATION OF REFINERY GASES.

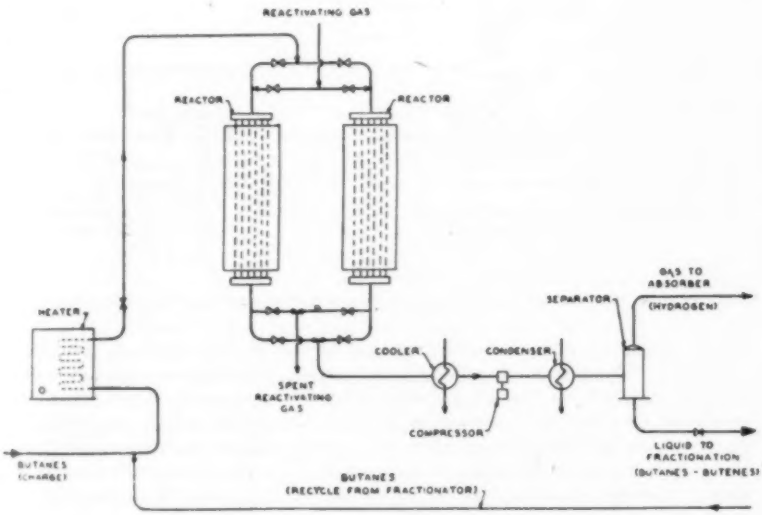


FIG. 10.

CATALYTIC DEHYDROGENATION PROCESS.

which causes the splitting off of two atoms of hydrogen from butane molecules to form butylenes, in accordance with the following equation:

$$\underset{\text{Butane.}}{H_3C{-}CH_2{-}CH_2{-}CH_3} \xrightarrow[\text{catalyst}]{\text{heat and}} \underset{\text{Butylenes.}}{H_3C{-}CH{=}CH{-}CH_3} + \underset{\text{Hydrogen.}}{2H}$$

The products are fractionated to separate hydrogen, butylenes, and unconverted butanes. The butanes are then returned for further treatment to bring about a high ultimate conversion to butylenes. Reactors are used alternately in processing and reactivating periods.

iso-*Octane Manufacture.*

A three-stage catalytic process that is used commercially to produce mixtures of eight-carbon-atom paraffins (octanes) of high anti-knock value from butanes employs, successively, (1) dehydrogenation of butanes to butylenes, (2) polymerization of butylenes to eight-carbon-atom liquid

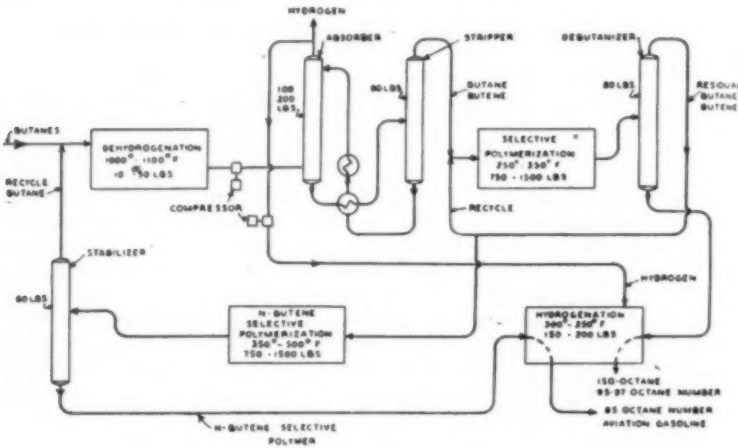


FIG. 11.

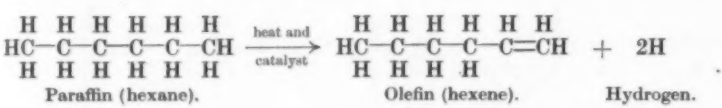
ISO-OCTANE PROCESS VIA DEHYDROGENATION–POLYMERIZATION–HYDROGENATION.

olefins (octenes), and (3) hydrogenation of the liquid olefins to paraffins (octanes). The process flow in a plant operating in this manner is indicated diagrammatically in Fig. 11, together with the temperatures and pressures employed in successive conversion steps.[6] Chromium oxide catalysts are employed in the dehydrogenation step, solid phosphoric acid catalyst in the polymerizing steps, and nickel catalyst in the final hydrogenation steps. Careful fractionation is employed to separate the unconverted materials for recycling to gain further contact with the catalysts, in order to assure the highest possible yields of the octanes. As indicated in the diagram, the highest octane-number product is produced from the more

readily polymerized olefin, *iso*butylene, which, on account of its branched structure, yields polymers of more highly branched structure than those produced from the normal or unbranched butylenes.

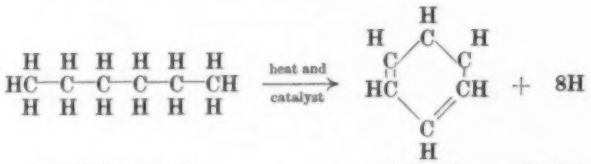
Hydroforming.

Several catalytic processes are used to convert open-chain saturated hydrocarbon constituents of gasolines into closed-chain hydrocarbons, and thereby raise their anti-knock values. When an open-chain hydrocarbon having at least six carbon atoms in a row is contacted with a dehydrogenation catalyst, such as chromium oxide, under mild conditions of temperature and for a short time, two hydrogen atoms may be split off, thus:



Paraffin (hexane). Olefin (hexene). Hydrogen.

If, however, higher temperatures and longer times are used, the removal of hydrogen rapidly progresses until eight hydrogen atoms are lost, and a compound of ring structure is formed, thus:



Paraffin (hexane). Aromatic (benzene). Hydrogen.

The formation of the ring-structure hydrocarbons corresponds to a great increase in anti-knock value, and so open-chain hydrocarbons or gasoline fractions containing them (and in consequence having knocking properties) may be treated by contacting them with chromium oxide catalysts at temperatures of the order of 1000–1100° F. to obtain hydrocarbons having higher anti-knock values. This process is known as *dehydrocyclization* or *cycloversion*. When the cyclization process is applied to certain gasolines or naphthas, the activity of the catalysts may diminish rapidly, on account of the separation of carbon or heavy tars accompanying extensive decompositions. As a means for overcoming this difficulty, it has been found that, if a certain amount of hydrogen is mixed with the vapours of the oils being dehydrogenated, the character of the decomposition reactions is modified and the activity of the catalyst is maintained over longer periods. This process, which is essentially reforming in the presence of added hydrogen, is known as the *hydroforming* process, and the usual process flow employed is indicated in Fig. 12.[3] Parallel reactors containing granular catalysts are used so that the mixture of oil vapours and hydrogen may be passing through one, while catalyst in the other is being reactivated by burning off carbon deposits accumulated during a

period of service. Hydrogen is separated and recycled, and the hydrocarbon products are fractionated into butanes and light and heavy liquid hydrocarbon fractions. The final solvent-extraction step shown in the diagram is employed to concentrate aromatic hydrocarbons when this is desirable. The hydroforming process is most frequently applied to straight-run gasoline fractions comprising principally paraffin and *cyclo*-

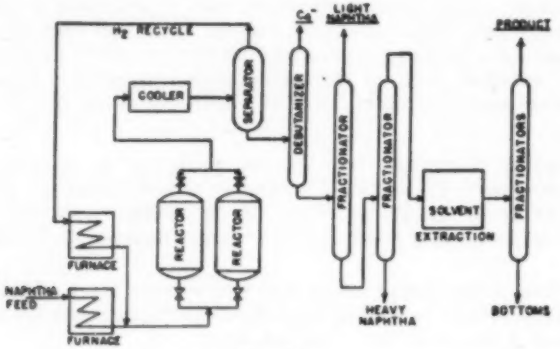
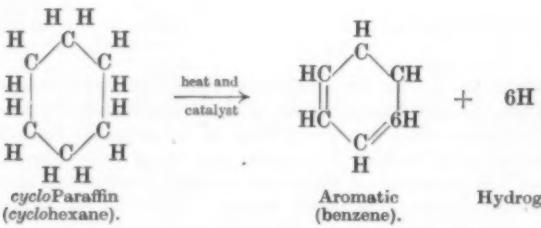


FIG. 12.

HYDROFORMING PROCESS.

paraffin hydrocarbons, and the principal reactions brought about involve the dehydrogenation of *cyclo*paraffins to aromatics as represented in the following equation:

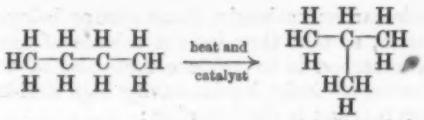


*cyclo*Paraffin (*cyclo*hexane). Aromatic (benzene). Hydrogen.

A certain amount of ring-compound formation occurs, however, as a result of the dehydrocyclization of the paraffins. The catalysts used in hydroforming processes usually contain molybdenum oxide.

Isomerization.

A means for producing gasoline of improved octane number from both hydrocarbon gases and hydrocarbon liquid fractions is the *isomerization* process, in which the structure of paraffin hydrocarbon molecules is altered in the direction of increased compactness. This type of hydrocarbon conversion reaction is exemplified by that occurring when normal butane is contacted with certain catalysts, such as aluminium chloride, thus:



"Normal" butane. "*iso*-" butane.

The normal, or straight-chain, structure of carbon atoms is changed by isomerization catalysts into an "*iso*," or branched-chain, structure, without any change in the number of carbon and hydrogen atoms in the molecule. The word isomer is also Greek, and means literally "same parts." Several advantages accrue from the transformation: (1) the boiling point is lowered; (2) the anti-knock value is improved; (3) the "*iso*" compounds are more reactive with other hydrocarbons, so that more hydrocarbons can be built up or synthesized from them.

The isomerization process is most readily utilized to convert normal butane into *iso*butane, and Fig. 13 shows a simple plant flow for the

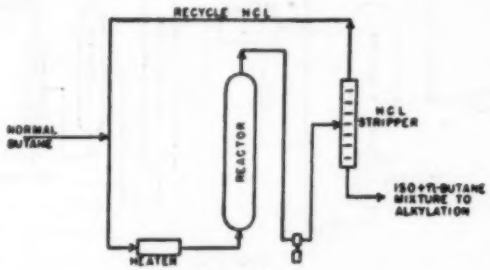


FIG. 13.

ISOMERIZATION OF *N*-BUTANE.

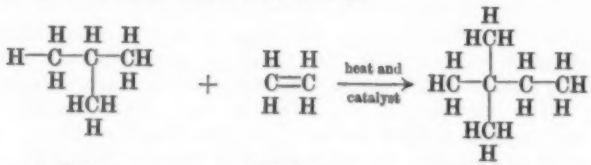
purpose.[3] Thus normal butane, separated from natural gas, still gases, or cracked gases by distillation, is passed through a heater to raise its temperature to about 250° F., and then passed through a reactor containing granular isomerizing catalyst. Hydrogen chloride (HCl) gas is supplied in small quantity to assist in the conversion.

The isomerization process can be used to a limited extent in the treatment of liquid hydrocarbon fractions to improve their anti-knock values. It can be applied best to low-boiling liquid fractions containing only saturated hydrocarbons—*i.e.*, paraffins and *cyclo*paraffins. When unsaturated hydrocarbons such as olefins and aromatics are present, even in small amounts, they may react with and damage the catalyst so that it ceases to function soon after the process has started.

Alkylation.

The principal value of the isomerization process is in the production of *iso*butane from normal butane, since the reactivity of *iso*butane permits its *alkylation* by olefins to produce liquid hydrocarbons of extremely high anti-knock ratings. The alkylation process involves the joining of alkyl

groups to a hydrocarbon molecule, these groups being paraffins minus one hydrogen atom, so that they have a valence of one. The effect of alkylating a hydrocarbon is to produce molecules of a highly compact structure and, correspondingly, liquids having high anti-knock ratings. A typical alkylation reaction is the following:



*iso*Butane (a gaseous paraffin). + Ethylene (a gaseous olefin). → *iso*Hexane (a liquid paraffin).

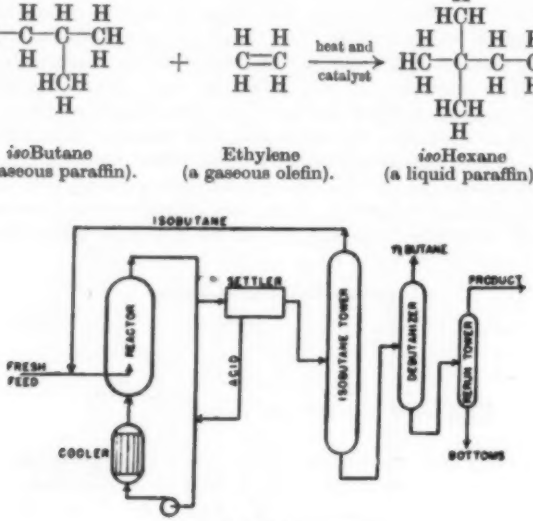


FIG. 14.
ALKYLATION PROCESS.

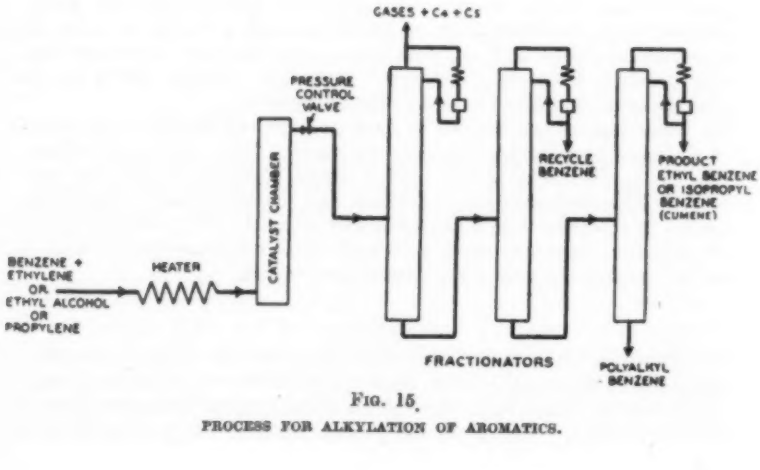
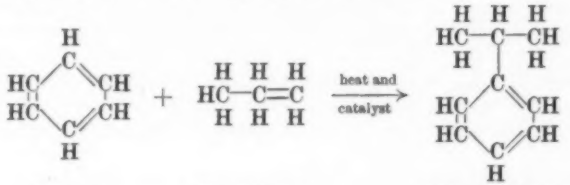


FIG. 15.
PROCESS FOR ALKYLATION OF AROMATICS.

Although *iso*butane can be alkylated by olefins at elevated temperatures without using catalysts, better results are obtained at lower temperatures when acids are employed to catalyze the reactions. Fig. 14 gives a simple process flow employed in alkylating *iso*butane to produce liquid gasoline hydrocarbons.[3] The feed is a proportioned mixture of *iso*butane and an olefin such as ethylene, propylene, or a butylene. A liquid acid is thoroughly mixed with the hydrocarbons in the reactor; the acid is allowed to separate from the hydrocarbons in a settler, and is returned to the reactor; and the hydrocarbon products are fractionated to recover unreacted *iso*butane for recycling, normal butane for isomerization, and the desired liquid *iso*paraffin product.

The alkylation process can also be applied to aromatic hydrocarbons to produce hydrocarbons valuable as motor-fuel constituents and in synthetic-rubber manufacture. A typical process of this character consists in alkylating benzene with propylene, thus:



Benzene. Propylene. *iso*Propyl benzene (cumene).

*iso*Propyl benzene has a very high anti-knock value in aviation gasoline mixtures and is better than benzene in such fuels.

Fig. 15 indicates a process flow applicable to the accomplishment of the above reaction.[7]

A mixture of an aromatic hydrocarbon, such as benzene and a compound capable of forming an alkyl group, is passed through a preliminary heater and through a catalyst chamber wherein the reaction of alkylation occurs. The products from the catalyst chamber are released through a pressure-control valve and fractionated in a stepwise manner. In the first step light gases are removed, in the second step unreacted benzene is taken overhead, and in the third step the desired alkylated product is recovered. The separated gases and the benzene may be returned to the primary stage of the process for further reaction.

The foregoing discussion has endeavoured to indicate the types af hydrocarbons which give the best anti-knock properties to a motor fuel and to develop briefly some of the processes employed in petroleum refining to produce the more desirable hydrocarbon types. In such a limited treatment it is not possible to include many details which are more or less common practice in the commercial operation of the various processes discussed. However, it is felt that the discussion will give some idea of the character of present-day petroleum refining processes which are aimed at improving both the quantity and the quality of internal-combustion engine fuels. Such processes are in a continual state of flux, and those that are in operation at the present time may become obsolete a few years

hence, owing to the rapid progress of chemical and engineering research. In any event it is certain that processes will always be available for producing fuels for whatever improved types of engines may be developed.

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[4] J. E. Bogk, P. Ostergaard, and E. R. Smoley, *Petroleum*, May 1941, p. 18.
[5] Geo. F. Fitzgerald, *Chem. Metal. Engng*, April 1939, **46** (4), 196.
[6] Berkman, Morrell, and Egloff, "Catalysis" (Reinhold Pub. Co.), p. 1031, dehydrogenation flow; p. 1035, *iso*-octane process flow.
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THE CORRELATION OF CETANE NUMBER WITH OTHER PHYSICAL PROPERTIES OF DIESEL FUELS.

Report by the Diesel Index Panel of Standardization Sub-Committee No. 4—Gas, Diesel and Fuel Oils.

Introduction.

The problem of correlating the cetane number of a diesel fuel with other physical properties is one of some importance, as has been recognized by the introduction of a considerable number of formulæ designed to permit the assessment of ignition quality. The present lack of general engine-test facilities serves to accentuate the desirability of obtaining a reliable relationship between cetane number and any of the more easily determined physical properties, such as aniline point, specific gravity, viscosity, and boiling range.

It is generally recognized that, as a rule, cetane number increases with increased paraffinicity, and since the physical properties mentioned above are all related to chemical structure, it was a logical step to look for some correlating formula.

The purpose of this paper is to report the work carried out by the Diesel Index Panel of I.P. Sub-committee No. 4. The Panel has reviewed such correlation methods or formulæ as have been put forward from time to time with a view to determining what degree of accuracy can be expected by the calculation of cetane number as opposed to actual engine determination.

Ignition Quality Formulæ.

The first accepted formula correlating ignition quality of a diesel fuel with other physical properties was the so-called "diesel index" (Becker and Fischer, *J. Soc. Aut. Engrs*, Oct. 1934, p. 376), which expressed a relationship between the product of the A.P.I. gravity and aniline point, and ignition quality.

$$(1)\ \text{Diesel Index} = \frac{\text{A.P.I. Gravity} \times \text{Aniline Pt. (° F.)}}{100}.$$

Since this relationship was formulated, a considerable improvement has taken place in the technique of cetane-number determination, and although diesel index is still widely used, it is recognized as being only a rough guide to actual ignition quality.

Since the introduction of diesel index, many other formulæ have been suggested, among which may be mentioned:

$$(2)\ \text{Calculated Cetane No.} = \frac{\text{Diesel Index} \times \text{Mid. Boiling Pt. (° F.)}}{790} + 13$$

(Esso Laboratories Research Division, Linden, N.J.).

(3) Esso Laboratories Research Division chart relating cetane number with A.P.I. gravity and mid-boiling point, or viscosity (Fig. 1).

(4) Calculated Cetane No. $= \dfrac{\text{Mid-Boiling Pt. (° C.)}}{5 \times (\text{S.G.})^4} - 56$

(Anglo-Iranian Oil Co.).

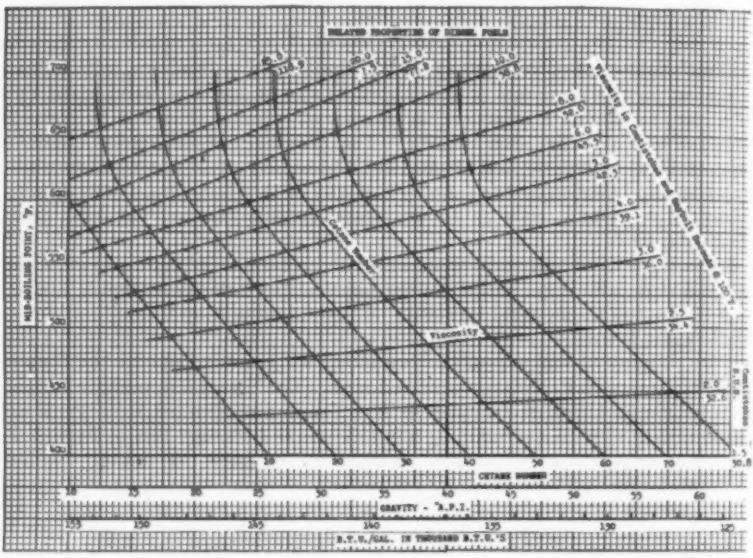

FIG. 1.

RELATED PROPERTIES OF DIESEL FUELS.

It will be seen that the above formulæ differ from the diesel index formula in that mid-boiling point is included—that is, the temperature in the I.P. distillation test at which 50 per cent. of the sample has distilled over.

Sub-Committee No. 4 of the Institute of Petroleum recently conducted an investigation into these and other correlation methods to determine with what accuracy cetane numbers could be predicted. At the same time it was hoped to produce an improved formula or chart which would give better results than any of the expressions hitherto suggested. The data on which this work was based were obtained on sixty-two samples which had been examined over the last three years in the Standard Inspection Laboratory's monthly diesel check sample programme, and these data were later extended by the inclusion of information concerning eleven samples tested by Shell, and ten by the Anglo-Iranian Oil Co. Nineteen samples on which data had previously been published by Schweitzer and Hetzel (*J. Soc. aut. Engrs*, 1936, **38,** p. 206) were also included.

A modification to equation (2) above was suggested by Esso European Laboratories, and this formula was also considered, viz. :

$$\text{Calculated Cetane No.} = \frac{\text{Diesel Index} \times \text{Mid-Boiling Pt. (° F.)}}{810} + 14.$$

A further modification which was included in the work was obtained by revising the chart shown in Fig. 1, the new proposed version of which is shown in Fig. 2.

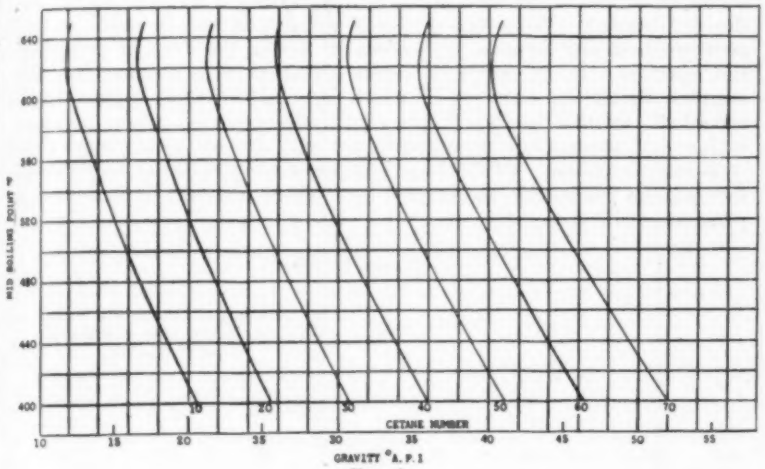

FIG. 2.

RELATED PROPERTIES OF DIESEL FUELS.

In connection with the work of Sub-Committee No. 4, the following formula was suggested as being simple and fairly accurate :

(5) Calculated Cetane No. = $\frac{2}{3}$ Diesel Index + 14.

This formula was found to give reasonably good results, but a modification, again incorporating the mid-boiling point, was found to be an improvement, and did, in fact, give the best results of any formula so far investigated :

(6) Calculated Cetane No. = $\frac{2}{3}$ Diesel Index + 0·068 Mid-Boiling Pt. (° F.) − 22
(Esso European Laboratories).

This formula can be applied graphically or by the use of the alignment chart shown in Fig. 3.

Even with this formula, however, in only two-thirds of the cases examined could the cetane number be predicted within ± 3 cetane numbers. In other words, for three cetane-number predictions made on fuels selected at random, it is to be anticipated that two will be between ± 3 cetane numbers, whilst the third result will be outside this bracket.

Consequent on the work of the Diesel Index Panel, Esso Laboratories Research Division evolved a further equation, again correlating cetane number with mid-boiling point and A.P.I. gravity in the following way:

(7) Calculated Cetane No. = 175·4 Log (Mid-Boiling Pt., ° F.) + 1·98 (A.P.I. Gravity) − 496.

This formula was applied to the data already considered by the Sub-Committee, and, as will be seen from Table I, 60 per cent. of the samples showed a calculated cetane number within ± 2 cetane numbers of the actual value, and 77 per cent. within ± 3, while only 6 per cent. fell outside the ± 5 bracket. While this degree of correlation was, in itself, an improvement on any formula yet examined, during the work of producing the equation (No. 7) estimated cetane numbers were obtained on 579 samples, the cetane numbers of which were known from engine determination. Of this number 70 per cent. gave estimated cetane numbers within ± 2 cetane numbers, whilst 83 per cent. differed by not more than ± 3 cetane numbers. Of this large number of samples tested, only thirty-five samples fell outside the bracket ± 5 cetane numbers, representing a percentage of 6 per cent.

TABLE I.

Formula.	Accuracy of Cetane No. Prediction; percentage within indicated bracket.		
	± 2.	± 3.	Outside ± 5.
C.N. = ⅔ D.I. + 0·068 M.B.Pt. (° F.) − 22 .	53	67	92
Modified Esso Lab. chart:			
C.N. v. ° A.P.I. and M.B.Pt. (° F.) (Fig. 2) .	45	67·5	92
C.N. = ⅔ D.I. + 14	45	60	87
C.N. = $\frac{\text{D.I.} \times \text{M.B.Pt. (° F.)}}{810} + 14$ . .	39	63	92
Esso Lab. chart:			
C.N. v. ° A.P.I. and M.B.Pt. (° F.) (Fig. 1) .	34	52	84
C.N. = $\frac{\text{D.I.} \times \text{M.B.Pt. (° F.)}}{790} + 13$ . .	23	5	82
Esso Lab. chart:			
C.N. v. ° A.P.I. and viscosity . . .	24·5	45	67
C.N. = D.I.	42	53	72
C.N. = ⅔ D.I. + 0·068 M.B.Pt. (° F.) − 0·685 ° A.P.I. + 10·5	41·5	61	91·5
C.N. = 0·18 × ° A.P.I. − 10·5 . . .	10·5	17·5	—
C.N. = 0·372 A.P. − 7 (A.P. = Aniline Pt. ° F.)	40	53	82
C.N. = $\frac{\text{M.B.Pt. (° C.)}}{5 \times (\text{S.G.})^4} - 56$ (Anglo-Iranian Oil Co.)	29	39	69
C.N. $\propto \frac{TA}{S^3}$ (T = M.B.Pt., A = Aniline Pt., S = Spec. Grav.)	34	54·5	80
C.N. $\propto \frac{TA}{S^4}$	32	59	84
C.N. = 175·4 Log (M.B.Pt., ° F.) + 1·98 (A.P.I. Gravity) − 496	60	77	94

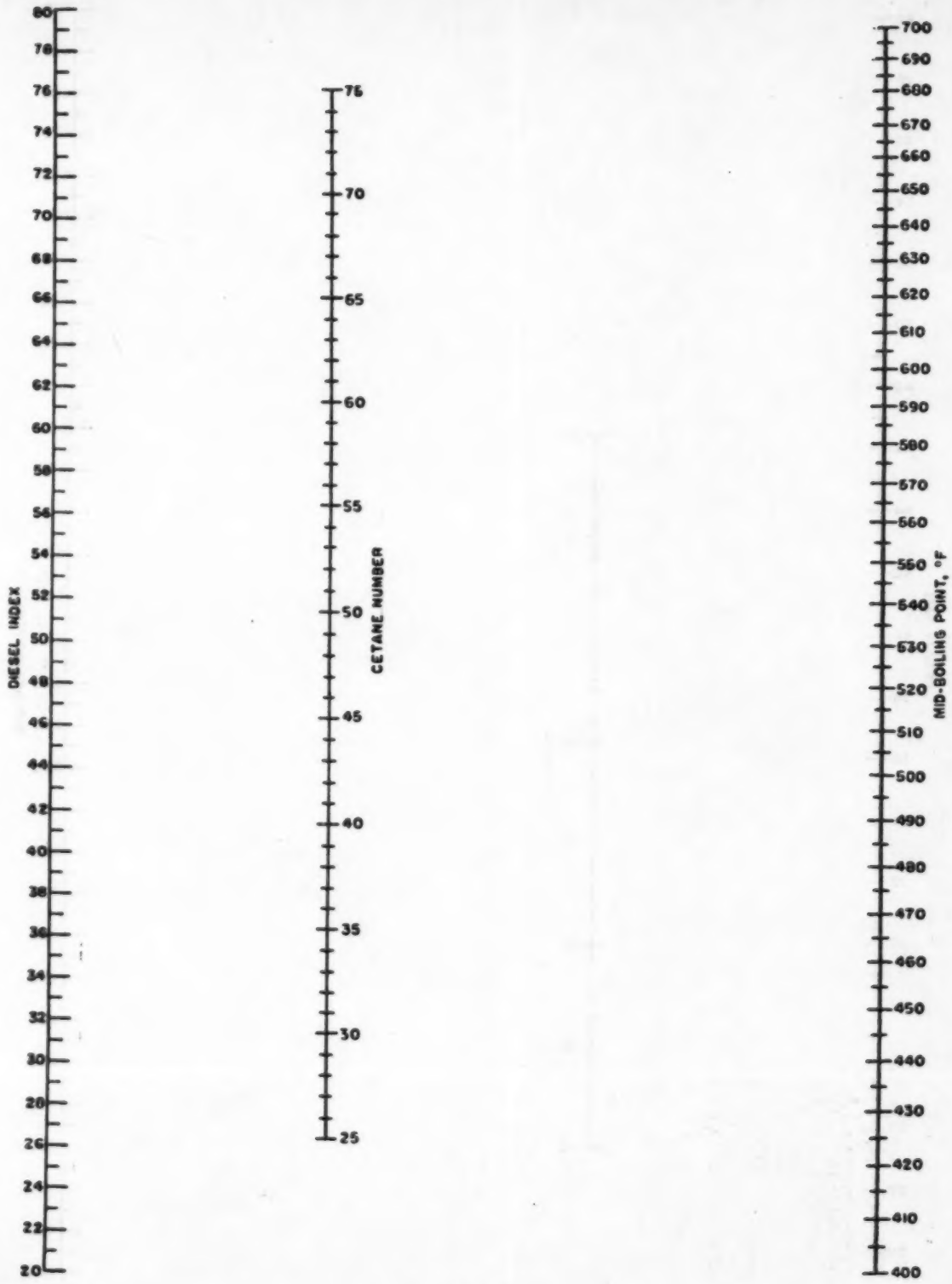

FIG. 3.

CETANE NUMBER–DIESEL INDEX–MID-BOILING POINT RELATIONSHIP.

Experience using equation No. 7 with diesel fuels prepared from shale oil or hydrogenated gas oil is very limited. With one sample of shale oil examined the actual cetane number was 57, which agreed exactly with the calculated cetane number. In the case of one sample of hydrogenated gas oil the actual cetane number was 48, the calculated cetane number being 51.

Conclusion.

On the basis of the work carried out by Sub-Committee No. 4, it is concluded that the most accurate formula investigated is :

(7) Calculated Cetane No. = 175·4 Log (Mid-Boiling Pt., ° F.) + 1·98 (A.P.I. Gravity) − 496.

An alignment chart based on this equation is shown in Fig. 4. The calculated cetane number is found by drawing a straight line from the A.P.I. gravity of the sample under test, on the left-hand side, to the mid-boiling point in ° F. on the right-hand side. The cetane number is indicated where this line cuts the cetane-number scale.

It should be noted that the above formula should not be used with oils containing ignition promoters. For the sake of brevity, it is suggested that the cetane number calculated by means of this formula should be known as the " cetane index."

THE 1944 EDITION OF THE I.P. "STANDARD METHODS FOR TESTING PETROLEUM AND ITS PRODUCTS."

By The Editorial Sub-Committee.

It is intended in this paper to review briefly the new edition of the I.P. "Standard Methods," in order to open a discussion on the subject. No attempt will be made to cover the changes in detail, since they have been fully dealt with already in the Report of the Chairman of the Standardization Committee, which is published in the book itself. Reference will be made only to the more salient modifications and innovations, and it is hoped that a vigorous discussion will follow, and that questions will be answered individually by the Chairmen of the various Sub-Committees concerned.

In the present revision the work has differed widely in nature from that of the 1942 edition. The 1942 book represented the first revision for seven years, during which period much of the machinery of Sub-Committees and Panels had become rusty and creaking, if not altogether motionless. In addition, the decision was taken to rewrite all the current methods according to a standard format. Most of the technical innovations then introduced were sponsored by the Editorial Sub-Committee, so we must, therefore, be forgiven for looking back with particular affection upon the 1942 book. In the case of the present edition, however, the work of revision has proceeded upon formal and correct lines, so that we cannot claim any technical responsibility. Phœnix-like, completely reconstituted Sub-Committees and Panels have arisen, and have been exceedingly active in the production of new methods and in the revision of old ones. Our work has therefore been restricted to its true function, which is, in the main, that of ensuring that new methods appear in the correct format, thus producing uniformity in the book as a whole, preventing anomalies which may arise when numerous Committees work almost independently of each other, and generally seeing the book through the press.

In our opinion, the most notable feature of the new edition is the extension of its scope. It is now the acknowledged intention of the Standardization Committee to cover, as far as is possible, not only methods for the whole gamut of products derived from petroleum, but also methods for materials which incorporate petroleum products and which are frequently analysed in petroleum laboratories. Thus a number of methods now appear for materials not catered for previously. Thirteen methods, sponsored by a new Sub-Committee, are included for chemicals derived from petroleum, three methods for bituminous emulsions appear, and one method for petroleum gases, whereas previously no method applicable particularly to any of these products was given. Thus the first steps have been taken in a development likely to become of major proportions in the future.

Since the methods for derived chemicals represent an entirely new venture, we shall refer to them first. The Sub-Committee, in its exploratory discussions, was faced with some difficult problems, and it decided to limit itself for the present to methods for chemicals derived from petroleum

and actually commercially available in this country. A number of chemical, physical, and technological tests have been produced for such products. The wetting-power test for detergents represents a tightening up of Drave's original procedure, and has proved of value in the development of a new synthetic detergent now being manufactured in this country. The method for surface and interfacial tension differs from du Noüy's original method by the substitution of a rectangular glass plate for the usual platinum ring, and thus avoids the intricate corrections which were sometimes necessary. In addition to its application to derived chemicals such as petroleum detergents, this method has been found very useful for some of the usual liquid petroleum products. Another method which should have a wide application is that for the determination of molecular weight by depression of the freezing point. Other material produced by this Sub-Committee comprises chemical methods for the acetate content of solvents, alkalinity of esters, ketones, etc., halogen content, iodine value, and peroxide value of ethers. The B.S.I. method for unsaponifiables in drying oils has been adopted and methods are given, in addition, for estimating the drying time of aromatic petroleum residues, the naphthenic acids in soda-neutralized oils, the residual odour of lacquer solvents, and the inorganic salts in alkyl sulphates. Thus it will be seen that a substantial effort has been made to provide analytical methods for the newer members of the petroleum family.

The Apparatus Sub-Committee represents another venture which has also started from scratch. It has carried out the considerable task of revising the whole of the thermometer and hydrometer specifications. The " war-time " specifications, published in this edition, have been produced in co-operation with the manufacturers, and although the new instruments are generally simpler in design and will be easier to make, the necessary accuracy has not been prejudiced thereby. A number of new thermometers has been specified, and this Sub-Committee has co-operated with others on general questions of apparatus revision.

The new sampling method is in some respects the most noteworthy of all the revisions in the book. This method, or really group of methods, has been completely rewritten and has been made more comprehensive, and it is hoped that the new arrangement will enable the modernized procedures for the sampling of the various petroleum products to be located rapidly by the user. A procedure for liquefied petroleum gases, utilizing a metal pyknometer, has been added to the comprehensive specific-gravity method. Only minor modifications were found necessary for the remainder of this method, which was considerably extended and completely rewritten for the 1942 edition.

The crude oil Sub-Committee has worked out a scheme, which appears as an appendix to the book, for the evaluation of crude oils. It includes an abbreviated scheme of inspection, which utilizes methods which were already in existence, and is adequate for routine examination, and a more detailed scheme which requires specialized apparatus and technique. The detailed evaluation has involved the adoption of a new vacuum fractional distillation method so that yields of desired cuts may be predicted. This method is considered to give results comparable to those given by modern refinery practice. A new method, of the conventional type, is also given

for the batch distillation of residual oils, but a single-flash pipestill method would be preferred, and will be adopted in the future, when the difficulties of standardizing such an apparatus and technique have been overcome. A method for estimating salt in crude oils, etc., has also been included.

New methods for light distillates are the water tolerance of motor fuels, which determines how much water may be added to an alcohol fuel before separation into two layers occurs, the estimation of tetraethyl lead by hydrochloric acid extraction, and the residue on evaporation of kerosines, etc. For the latter estimation the A.S.T.M. method for gum content of gasoline is used, except that the temperature of evaporation is higher. A third procedure involving the use of a sealed bulb has been added to the aniline-point method. Important modifications have been made to the cold test for motor fuel, colour by means of the Lovibond Tintometer (the B.D.H. pattern instrument having been made obligatory), Abel flash-point, and gum (existent) in motor fuel. In the oxidation stability test for gasoline (formerly called " gum " stability) the definition of " break-point " has been amended, and the Reid vapour-pressure method has been extended to cover fuels containing water-soluble constituents.

The Sub-Committee for gas and fuel oils, etc., has introduced a method for the low-temperature filtration of gas oils, etc., which provides a simple means of determining the tendency of fuels to clog gauzes at low temperatures. An important modification has been made to the wording of the introduction to the diesel index, to make it quite clear that there is no direct correlation with cetane number. A number of fuel-oil stability tests have been under investigation, and although not ready for the present book, it is anticipated that two or three tests of this nature will be standardized in the next edition.

In the field of engine-testing the main modification has been the elimination of the use of a throttle plate in the knock-rating method for motor fuels. New secondary reference fuels have been calibrated for aviation-fuel testing. Consideration has been given to the existent mechanical tests for lubricants but so far no test has been selected for standardization.

Turning to the work of the lubricating oil Sub-Committee, a major modication has been made to the absolute viscosity method, and it is now made clear that the total permissible error is $\pm 0{\cdot}6$ per cent., this of course having an appreciable influence on the magnitude of the possible error in viscosity index. 40 per cent. sucrose solution is no longer allowed as a primary viscosity standard, whilst the types of instrument permitted are now only the three described. A straight-necked flask has been permitted as an alternative to the Kohlrausch flask in the Redwood method. For the determination of acidity, alkali blue may be used as an alternative to phenolphthalein, and will be found a superior indicator for dark oils. Modifications have been made to the methods for ash content, Ramsbottom carbon residue, diesel-fuel diluent, dielectric strength, sludging value of transformer oils, and oxidation test for lubricating oils. The method for estimating free acid and free alkali in greases has been substantially altered to cover cases in which both free acid and alkali are present together, whilst for the drop-point of greases the B.S.I. apparatus has been taken over with the recommendation that the metal cup be used.

The asphaltic bitumen Sub-Committee has sponsored a new method, that for the determination of breaking point (Fraass), which indicates the temperature at which a film of the bitumen first cracks when subjected to a specified rate of strain. The decision having been taken to specify methods for the examination of bituminous emulsions, a beginning has been made with methods for residue on sieving, coagulation of binder and long-period storage stability, and with the extension of the water-content and sampling methods to cover emulsions. Although no methods have been standardized in connection with the use of petroleum products in soil-stabilization processes, much work on this subject has been accomplished. For the present it has been decided to publish the recommended methods in the *Journal*, and to reconsider them later in the light of further experience. It is anticipated that they may in the future make a significant extension to Standard Methods. For the asphaltic bitumen penetration test the A.S.T.M. needle has now been specified, and for the ring-and-ball softening point the tapered ring, with the proviso that the straight ring or the A.S.T.M. new shouldered ring may be used, if preferred, for non-referee tests.

A new and simple method, using a rotating thermometer, has been introduced for determining the congealing point of petroleum waxes. This will give a satisfactory result with all petroleum waxes, and hence the scope of the setting-point method has been limited to paraffin waxes and scales, the procedure formerly specified for difficult samples having been deleted. Although no method for the determination of oil in wax has been standardized, the need for such a method is recognized, and the search for a simple and accurate procedure goes on.

A few of the methods which appeared in the 1942 book have been deleted through obsolescence. These are the potential gum in motor fuel, asphaltum (soft asphalt) in asphaltic bitumen and distillation of crude oil (the A.S.T.M. 300 ml. Hempel method).

It is the wish of the Standardization Committee to reduce the present multiplicity of official petroleum tests and to work towards the ideal of a single collection of official Anglo-American test methods. As a step towards such co-ordination, the Chairmen of the various Sub-Committees have corresponded with their opposite numbers in the A.S.T.M., and, as the present edition testifies, wherever possible A.S.T.M. and B.S.I. methods have been adopted without or with the minimum of modification. However, progress has been slow and it is necessary to do a great deal more towards this end, which is so desirable in view of the close coupling of the British and American petroleum industries.

In concluding this brief review we wish to thank all those unnamed members of the petroleum industry who have assisted us in the preparation of the new edition.

INSTITUTE OF PETROLEUM.

A MEETING of the Institute of Petroleum was held at Manson House, 26, Portland Place, London, W.1, on Friday, 24th March, 1944, at 4 p.m. The PRESIDENT, PROFESSOR F. H. GARNER, was in the Chair.

MR. CANTOR read the following paper:—

"The 1944 edition of the I.P. Standard Methods for Testing Petroleum and its Products." By the Editorial Sub-Committee. (See pp. 198–201.)

DISCUSSION.

The PRESIDENT said that the subject for discussion was the new edition of "Standard Methods of Testing." He wished to express the appreciation of the Institute of the work of the members of the Committee, particularly that of the Chairmen of the various Sub-Committees. A closer liaison with the A.S.T.M. D.2 Committee was being effected, and the Chairmen of the Sub-Committees could, and usually did, get in touch with the Chairmen of the Committees of the A.S.T.M. The most valuable work was done while the methods of test were in process of being drawn up, rather than when they had been published. In this tribute he wished to associate all the members of the Sub-Committees and the Panels and working groups. It said a great deal for their energy and devotion that so many men working in the laboratories of the oil companies and Government laboratories were able to devote so much time to the work under present conditions, even although nearly all the work was directly related to problems arising out of the use of petroleum products for war purposes.

When the Sub-Committees had completed their work the Editorial Sub-Committee prepared the methods for publication. They had on previous occasions expressed their indebtedness to the Editorial Sub-Committee for this work, which could only be adequately appreciated by those who were in close contact with it. For the new edition, a number of new test methods had been incorporated. The Editorial Sub-Committee had made a number of valuable suggestions as regards the form for drawing up methods of test and such matters as the type of illustration to be used. These had been adopted, and would be put into force as and when the opportunity offered. It was said that no book was free from errors, and in the present book there were some corrections to be made. One was to rectify the omission of Dr. Howes' name from the list of members of the Standardization Committee. It was very desirable that certain of the methods should be in complete accord with the A.S.T.M. methods, and alterations had to be made in the Knock Rating of Motor Fuels, I.P.—44/44(T) consequent on the omission of the throttle plate. A revised edition of this method of test in line with A.S.T.M. method D357–43T would be published at an early date with the *Transactions* of the Institute. Members had had an opportunity of reading the report of the various Sub-Committees, and he hoped it might be possible to keep the Institute even more closely in touch with the work of the Standardization Committee, perhaps by reports in between the annual reports when occasion justified it.

One new development was the Sub-Committee on Nomenclature, under the Chairmanship of Dr. Broom. The object of this Sub-Committee was to draw up definitions which might be of general use. These were not intended to be legal definitions, but there had been a general demand for some simple definitions even in the case of primary products; in particular, this demand had come from the users of the products concerned.

He could not close these few remarks without expressing the high appreciation which was felt by the Standardization Committee for the work of the staff of the Institute, particularly of Miss Gilley.

MR. J. S. JACKSON opened the discussion and emphasized that the Standardization Committee were genuinely anxious to have criticism of the "Standard Methods." Criticism was always stimulating, and was an indication of life and interest: in fact, progress in such matters as the development of standard methods mainly arose from criticism.

The introduction of tests for bituminous emulsions constituted something of an innovation. In this connection there had been effective co-operation with the Road Emulsion and Cold Bituminous Roads Association, which would ensure that these emulsion methods would be generally acceptable.

The report of the Soil Stabilization Panel would appear in the near future and would, he hoped, constitute a valuable contribution to our knowledge of the subject.

In considering this new edition, efforts had been made to eliminate obsolete methods, and he stressed the importance of a systematic pruning if the volume were not to become unwieldy.

Careful consideration had also been given to the question of alternative methods, and the Committee were of the opinion that, where rapid alternative methods were in general regular use, they should appear in the book along with the Standard "Referee" methods. The aim was to produce a volume which would contain all the "Petroleum" methods used in any well-equipped petroleum laboratory. With this aim in view, more methods which at present were scattered throughout other publications, such as B.S.I. Specifications, were being collected with a view to publishing them in the next edition of Standard Methods.

He regarded the inclusion of reports from the various Chairmen of Sub-Committees as a very useful development, providing the reader with an effective guide to additions and alterations.

Mr. Hurt said that he had taken the opportunity of running through the new edition alphabetically, and selecting those parts which particularly interested him.

He drew attention to the alternative method for acidity, employing alkali blue in place of phenolphthalein. This alternative method had been known to him for some time, but although probably the better method, it had not been employed by him as a general rule, for the following reason.

Acidity figures were collected and recorded by various people over a term of years. He had felt that use of a slightly different method might lead to slightly different results being obtained. In the event of discordance arising from this or any other cause, explanation would be made difficult. The phenolphthalein method had had the merit that it was the one sponsored by the Institute of Petroleum.

The method for aniline point recommended that "each sample shall be dried by shaking with anhydrous calcium chloride, or sodium sulphate," but no length of time for shaking was given. In an earlier edition of the book, the sample was prepared by standing over calcium chloride for seven hours at 120° F.—an inconvenient procedure. As he felt that both anhydrous calcium chloride and sodium sulphate were somewhat slow in their dehydrating action, inclusion of a minimum time of shaking seemed to be desirable.

The method for hard asphalt was a little vague as regards the quality of the filter-paper. Whatman No. 40 was suggested, but not definitely laid down. Some years ago, following variation of his results from similar ones determined abroad, he decided to use No. 44 Whatman filter-paper, which had the property of retaining the very finest precipitates. Thus his results would tend to be higher than those obtained by using a paper of less retentive properties. It had occurred to him that a Gooch crucible could well be used in place of filter-paper, except that by its use, and in the absence of solution in benzene, material other than asphalt not soluble in 60–80 petroleum spirit would be recorded as asphalt.

Method I.P. 83/44 "Halogen Content Determination (Proposed)," was disappointing in that it referred only to "nearly pure organic halogen compounds derived from petroleum," and not to halogenated compounds present as additives in mineral oil. With the advent of heavy oil containing additives, such as 10 HD and 30 HD, the development and standardization of methods for detecting and possibly estimating them were desirable. Recently he had had occasion to estimate the phosphorus in an HD oil, and found that the combustion of the usual 1 gm. in a Mahler Bomb, in conjunction with a colorimetric method for phosphorus determination, was satisfactory.

Under the Air Ministry Oxidation Test he was somewhat surprised to note the omission of a method for calibration of the manometers. The old DTD 109 specification gave a method which, if cumbersome, was satisfactory. The present book gave no guidance in this direction.

In regard to the method for determining the unworked consistency of greases,

he emphasized the point of employing a tin of adequate diameter where soft greases were concerned. He added that the American pound tin, being usually different in diameter from the English variety, could lead to discordance.

MR. E. A. EVANS said that alkali blue was introduced to overcome the difficulty of end-point when using phenolphthalein. When the free acidity was low, as in unusued oils, there was usually no difficulty with phenolphthalein, but when the acidity rose, as in the case of used oils, it was much more difficult to obtain a sharp end-point with phenolphthalein. It might be suggested that acidities of 1·0 and over had nothing whatever to do with new oils and should not be catered for in "Standard Methods." Admittedly "Standard Methods" did apply essentially to unused materials, but it was hoped that some of the methods at least could be utilized for used products of petroleum.

So far as was known, the two indicators gave precisely the same acidity reading, although there was a bigger spread in the results when using phenolphthalein than when using alkali blue. It should be noted that when using alkali blue the oil was treated with a mixture of benzene and alcohol, but when phenolphthalein was used, only alcohol was added.

The halogen test was introduced into Standard Methods for nearly pure halogen containing organic compounds. The time was not quite ripe to standardize a method for the estimation of halogens in oils containing additives. He thought that the number of additives used in lubricants was comparatively small numerically. Of course the number which had been suggested in the literature was enormous. Perhaps it was that comparatively few of the compounds which had been suggested had survived practical trials.

The estimation of phosphorus had been fairly well agreed by those people who were dealing with phosphorus containing additives. For estimating tributyl phosphite, the zinc oxide method was usually accepted. Some people had difficulty with it, but in general it was reasonably satisfactory, and it might be accepted later as a Standard Method.

The estimation of sulphur-containing additives was complicated by the presence of naturally occurring sulphur in the oil. Frequently, the amount of "additive" sulphur was a good deal less than natural sulphur. Therefore, other methods must be adopted to ascertain whether sulphur additive is present.

Organo-metallic additives were being used extensively. It should be a fairly simple problem for a chemist to estimate the content of metal by orthodox procedure.

The estimation of unworked grease penetration was not regarded very favourably nowadays, owing to the difficulty of obtaining a sample in the unworked condition. Influences were at work to condemn the use of the measurement of the unworked consistency. Of course there might be cases where it would be an advantage to know the value of unworked consistency, but these cases should be regarded as exceptional.

The calibration of the air flow in the oxidation test was now left to the personal choice of the individual operator. There were a number of very well known methods, therefore there was no special reason why one should be selected as standard.

DR. E. B. EVANS said that Mr. Hurt had directed attention to a point which had perhaps been insufficiently standardized in the Aniline Point method, and he would direct the attention of Sub-Committee No. 3 to it.

MR. J. S. JACKSON said that there had been some misunderstanding about alkali blue. He had now ascertained that the grade available in this country was 6 B, and it had been shown that this grade was entirely satisfactory.

MR. EMERY desired to congratulate the Institute on the adoption of alkali blue, although in fact it was nothing new, having been suggested by Lewkowitsch at least as far back as 1904. He asked for comments on the test for saponifiable matter. Surely one test would cover both saponifiable and unsaponifiable matter. As far as the volumetric method was concerned, he was afraid that when he first started he used absolute alcohol as the solvent material, but it did not take him very long to discover that much the same result was forthcoming if industrial methylated spirit was used. During the last ten years he had been using industrial methylated spirit, and

he would be interested to learn if others had done so. It might be an alternative method. The boiling time was stipulated as three hours, but saponification of straight fatty oils was virtually complete in five minutes, certainly in a quarter of an hour. It would be interesting to have an opinion on these points.

So far as the estimation of halogen was concerned, frankly he disliked a method which involved a piece of special apparatus used for that estimation only. He had been using a method which seemed to him satisfactory—namely, the use of a fusion with two parts of potassium hydroxide and one part sodium peroxide, and asked for comments.

MR. E. A. EVANS said that the alcohol which was available years ago was not sufficiently good for saponification values. It tended to go brown very quickly, consequently one had to resort to better-class alcohol, and the only one which was available in those days was absolute alcohol. Of course, things had changed in recent years; a very good industrial alcohol was now available, and was sufficiently stable to caustic potash for saponification values. It might be proper to suggest that the term "industrial alcohol" might not define the solvent sufficiently accurately. Therefore, the chemist must take such precautions as he considered necessary. It was true to say that many pure fats could be saponified in the course of a few minutes, but of course there were some which were not so readily responsive. The reason why boiling for three hours was incorporated in "Standard Methods" was to ensure that a very viscous oil containing as little as 2·5 per cent. of fatty oil would be completely saponified. If a particular chemist was dealing exclusively with a mineral oil containing, say, 30 per cent. of rape oil, he was probably justified in boiling for only half an hour. Standard Methods were intended to cover as wide a field as possible.

Whilst it might be true that alkali blue dated back to 1904, it must be remembered that the manner in which we were using alkali blue in 1944 was somewhat different. The principal difference was that we sensitized the indicator immediately before use.

MR. EMBRY said that as far as the saponification value was concerned, if one saponified for a very long boiling time the net result was increased attack by caustic on the glass, so that what one lost on the swings one gained on the roundabouts by long boiling. What was wanted in order to make this method really good was a perfectly alkali-resistant glass.

MR. E. A. EVANS said that there was really no difficulty. Any error which was due to the action of the alkali on the glass was taken care of in the blank estimation.

MR. CANTOR said that he did not speak from experience of the particular fusion method mentioned by Mr. Emery, but similar fusions with lime and magnesia and so on were likely to give low results owing to loss of volatile chlorine compounds and that might be a difficulty.

MR. E. A. EVANS said that he had used the peroxide method for a number of years, and had found it perfectly good, but it depended entirely, like all these fusion mixtures, on the care with which the product at the bottom of the vessel was covered. The original method was published in the *Journal of the American Chemical Society* about 1908.

MR. BEECROFT had something to say from the point of view of the user.

When the 1942 edition was published it was regarded as a very welcome step forward, and the close collaboration between I.P. and A.S.T.M. was particularly gratifying, since his Company had prepared a series of standard methods taken from I.P. third edition and the A.S.T.M. However, when the 1944 edition was about to be published, fears were expressed as to how far this would effect these methods, and even how far these new 1944 methods could be considered as a stable basis in view of the proposed annual publication of "Standard Methods." This annual publication was thought to be unwise from the users' angle where oil testing was an auxiliary subject, but if the present procedure of directing attention in the Foreword to the alterations and additions in the Report of the Standardization Committee is continued, then the user would only have to turn to the first few pages to learn where the modifications lay.

The new edition had only come to hand that afternoon, and so reactions were

necessarily incomplete, but the following items were commented upon. The question of acidity had been raised, and he agreed that phenolphthalein, though very useful, was not completely successful for dark used oils, and stated that the indicator phenol-tetrachlorphthalein had been used in one of their laboratories with success. The *p*H range was very close to that of phenolphthalein, and the colour change was good, although it was mentioned that no actual black oils had been used.

The smaller crucible now recommended for ash contents was in use in their laboratories, and gave very good results. A "Drip Rod" was included with the tared beaker plus oil. He asked why 1·0 N alcoholic potash was preferred to 0·5 N for saponifiable matter (Method A), since the ultimate concentration was 0·5 N. There had been little trouble with the keeping properties of 0·5 N, although possibly for solutions kept over prolonged periods 1·0 N would be preferable. In agreement with other speakers, this was an example of where industrial alcohol was used, and this use he believed to be a fairly common practice.

It was suggested that with specific-gravity determinations using the drilled-stopper specific-gravity bottle, the method was speeded up and made more accurate when, (*a*) the liquid was put into the bottle at the temperature at which the gravity was required and then allowed to stand in the bath for only five minutes, and (*b*) the bottle was then chilled very slightly and the capillary height fell ½ to 1 cm. This latter precaution ensured no evaporation loss in the subsequent thorough drying and weighing of the bottle, particularly when volatile liquids were used. Also there was no mention in the new edition of a method for determining the gravity of water-soluble products, and the flotation method for solids lighter than water did not appear to be particularly good—what was wrong with the displacement method using a sinker?

In using the Redwood instrument he questioned the practicability of the very lengthy preheating time, particularly when a temperature of 140° F. was required. Viscosity determinations in their laboratories were now almost solely confined to the Fenske viscometer, and the I.P. permitted an increased range to that allowed by the A.S.T.M. for the various tubes, but insisted upon greatly increased minimum preheating times by, in some cases, 400 per cent. The A.S.T.M. minimum times had been used as a basis and satisfactory duplications obtained. The method he used was to commence the first run in line with A.S.T.M. time limits, and frequently a second or duplicate run with the same instrument and the same oil had been finished before the I.P. time had elapsed. Should there have been any discrepancy, a third run could then be carried out, which would then be in accordance with I.P. times.

A rather curious anomaly concerned the apparatus for water evaluation, in that it was only the 10-c.c. receiver which had a stopcock. Why was this so?

Confirmation was given of the fusion test for halogens, previously referred to by other speakers, and that led to a request for specific inhibitor tests which the users could apply so that they would be able to classify the inhibitors without going too deeply into the identification.

The question of doping had now become increasingly important, and surely it was not unreasonable for the consumer to know to what types these dopes belonged, so that he could check his supplies and be satisfied that they were uniform. This particularly applied when oils were used which gave very satisfactory results. A second point was that a strong feeling existed that a number of these tests were tests which the industry had now outgrown and the uses for which were now out of date. Even fluid film lubrication was a case where the viscosity did not nowadays appear to bear the same relationship to the practical working of the oil, as it had in the past. This was particularly so since dopes had become prevalent, and petroleum technologists were asked if it were not possible to produce a simple machine which would give direct fluid lubrication values.

The question of boundary lubrication had been dealt with satisfactorily, and he felt that if fluid lubrication had been a new field, then the viscometer would not have been the final piece of testing apparatus.

Another illustration of an out-of-date test was the flash-point determination; for most purposes this appeared to mean very little, and were there not, in fact, other tests which had outstripped their usefulness? The consumer carried out these tests, but was he not, in fact, wasting his time?

MR. MASKELL said that the first speaker, Mr. Jackson, had mentioned the desirability of eliminating obsolete methods of test. He himself thought that a number

of tests could be cut out. If we were going to keep adding new methods, revision of the old was necessary, and methods which were not now in use should be deleted. He was glad to hear Mr. Jackson making the point he had, with regard to the retention of certain alternative methods of test. He thought it was important that some of these which were in general use should be retained for a time, to give the user an opportunity of experimenting before switching over from his present method to the new standard. As regards the new method for determination of acid value, a lot of practical work had been carried out by the Panel before the introduction of the alkali-blue indicator was finally decided upon. When such a new method of test as this had been adopted, and had been found to give general satisfaction to users, then was the time for the old method to go out, but until then the original method should remain as an alternative.

The question had been raised regarding the oxidation tests, and whether a standard method for calibration of the air gauges should not have been given. In fact this point had been discussed, but it was purposely omitted, because there were a number of suitable ones available for carrying out the calibration, and it was not considered necessary to specify a particular method. Provided the result was the same—that is to say, provided that the air flow was correct—it did not matter what method was used to determine it.

Several speakers had mentioned the test for saponification value and thought that this called for some revision. He himself believed that there were several points about this method which could with advantage be looked into, but in justification of the Panel dealing with this test, as with certain other tests for lubricating oil, it should be stated that time was the limiting factor, and that the work of the Panel had been such that they had up to the present had no opportunity of considering the method for saponification value. With regard to the use of phenoltetrachlorphthalein as indicator, this had not been tried, although the Panel did experiment with a number of other possible indicators, eventually coming to the conclusion that alkali blue was by far the best for all types of oil, although phenolphthalein was fairly satisfactory in certain cases.

Dr. Anfilogoff said that on the question of additives Mr. Beecroft had suggested that the customer wanted to be sure that he was getting value for money. It seemed to the speaker that this was a matter where some measure of safeguard was required by the petroleum industry, and he did not see the necessity for formulating tests for the additives the oil companies, after a great deal of research, had seen fit to put into the oil. As far as he knew, the oil companies did not, as yet, go to the user and sell oil on the basis of price versus additive content. If, to satisfy the user, they had to provide him with information that the additive was of a given elemental chemical analysis, or was a defined chemical compound, this would mean little to the user in determining the value he was receiving, unless he was able to undertake a similar extensive range of engine tests to those which the supplier himself had undertaken in order to justify the inclusion of the given additive in his oil. Indeed, some harm to the industry might result if the precise formulæ of the given additives required to be common knowledge. This seemed to be a case where the industry could satisfactorily be left to give of its best to its customers.

Dr. Anfilogoff said that he had understood a previous speaker to declare that the indicator suggested in the place of phenolphthalein was proposed because it gave more accurate results on used-oil acidity determinations. He could not really visualize a set of service conditions where laboratory accuracy of the order suggested was required on a used oil. He had not been able quite to follow the point.

Mr. Beecroft said that he had put it forward as an indicator satisfactorily used in some of their laboratories. Surely the inclusion of alkali blue meant that a greater accuracy was sought. One further point was that a lot of tests had now to be done by relatively unskilled people, and it was far better to have something which gave the most pronounced end-point. Incidentally they favoured the A.S.T.M. method of acidity.

Mr. Maskell said that they employed the A.S.T.M. method for used oils in his laboratories, but although this was perfectly satisfactory for the purpose, he did not think it was as good as the new I.P. Standard Method for unused oils.

The CHAIRMAN said that two points remained still unanswered, one of them in connection with specific gravity, and the other the water estimation.

MR. J. ST JACKSON said that the Apparatus Sub-Committee had not yet dealt with the Water Estimation methods. They hoped to overhaul and possibly simplify the apparatus specification considerably.

MR. BEECROFT stated that when they found oils which had 1 per cent. of such and such an additive giving good results, they wanted to be certain further supplies were uniform in their inhibitor content.

DR. ANFILOGOFF replied that he thought, almost certainly, the supplier could be trusted in a case like that.

DR. E. B. EVANS said that he thought Mr. Beecroft preferred the method of chilling the gravity bottle to prevent loss by evaporation, but the best method with volatile materials was to use the Regnault bottle, and avoid evaporation that way. He did not think there was any particular advantage in using the drilled-stopper bottle for volatile products. It depended on the accuracy required, but the hydrometer usually gave sufficiently accurate results. The point with regard to soluble products had not been considered, but was it necessary to determine the gravity of greases which were appreciably soluble in water? As regards the flotation method for waxes, he personally might not wish to use the method, but it was quite widely used for routine examinations in some laboratories. He wished to emphasize Mr. Jackson's remark that criticism of the methods was very welcome, and that this applied not only to the methods published in the Handbook, but to proposed methods published in the *Journal*, and to suggestions for standardizing new methods. A few of such proposed methods had already appeared in the *Journal*, and a number more would doubtless appear in the future.

The size of the Standards Book had increased very much in the last two editions. Sub-Committee No. 3 had quite a lot of work on hand, including determination of mercaptan content, hydrogen sulphide content, and so on, and the same remark applied to other Sub-Committees. There was, however, the risk of over-standardizing, and this could be a great disadvantage. Analysis was, after all, an art as well as a science, and methods were capable of continual improvement. Premature standardization restricted such development and improvement. The co-operation of the user was required to improve "proposed" methods before they got to "standard" methods, and this applied especially to those methods which were published in the *Journal*, but had not yet got to the stage of "proposed" methods in "Standard Methods." Criticism of such methods, and also suggestions as to other methods which should be standardized, would be appreciated. There was sometimes a strong temptation to include methods which were in fairly wide use, but on which further comment from the industry as a whole was very desirable, before their inclusion.

MR. E. A. EVANS said how much he had appreciated the remarks from the visitors. It was very helpful indeed to have criticisms from people outside the Petroleum Industry. It would be a pity to let them go without dealing with some of the points they had raised. One speaker had referred to the difficulty of determining the specific gravity of those greases which were wholly or partly soluble in water. What useful purpose was to be served by attempting such a determination? All the soda greases were more or less soluble in water, consequently an emulsion would immediately be formed. He begged him not to waste his time, or that of the people he was trying to train, by introducing tests which were of no immediate value.

Apparently one visitor did not readily associate viscosity with film lubrication. The two were very closely allied, and could not be dissociated. Boundary lubrication, of course, was quite different, and could be dissociated from viscosity.

He thought that Dr. Anfilogoff had sufficiently stressed the position of additives. The time was not really ripe for the user to bother his head about the actual composition of an additive. There was a good deal of necessary secrecy about the composition of certain additives, and it was hardly to be expected that the industry would relax its secrecy in the interests of curiosity. The user was not really in a position to benefit very greatly from a knowledge of the particular additive, because it would

be impossible in the short time at his disposal to carry out sufficient investigation to prove its merits. For the time being users must accept gracefully the reputation of their suppliers that certain additives had certain properties. It would not be very long before the user had sufficient practical experience with a particular lubricant to prove that it had special qualities.

Dr. Anfilogoff pointed out that there was one fundamental difference between their method of producing the Standard Methods of Test Book, and that of the A.S.T.M. Committee, in that the A.S.T.M. Committee D.2 had produced, in conjunction with their Standard Methods of Test, a booklet, as far back as 1934, entitled "The Significance of Tests of Petroleum Products," which booklet contained therein, not only the scope of the test, but what was equally important, what that test meant to the user. He ventured to suggest to Dr. Evans that rather than increase the size of the present book, that they should stop short with what they had got, add, where necessary, to the scope of the tests, and, at the same time, provide the significance of the given tests to the user thereof. Provision of the significance of the tests would be doing the industry a far greater service than by offering in the next edition an extra fifty tests.

Mr. H. Hyams desired to comment firstly on the general question of criticism of the methods as they appeared both in the 1944 and the 1942 editions. He wondered whether they were going to get the maximum benefit from the comments which were likely to come forward at a meeting such as this almost immediately following the issue of the book. Criticism and comment would most probably come from people abroad, who did not have an opportunity of studying the methods for some months after they appeared; and if these two books were produced hard on each other's heels, containing a great deal of relatively new matter, they would not be likely to get the repercussions for some little time to come.

Another point raised by one speaker had been partly dealt with by Dr. Evans, the Chairman of the Panel responsible for specific-gravity methods. However, as the Chairman of the Sub-Committee which covered that Panel, he, Mr. Hyams, desired to approach the subject from a slightly different angle—namely, from that of quantitative measurement of bulk oil. He not only agreed with Dr. Evans regarding the futility of any sort of specific-gravity test for grease, but was strongly opposed to refinements of methods which, in fact, were meaningless. The purpose of the specific-gravity test, qualitatively, had very little meaning in these days, whereas twenty or twenty-five years ago one did associate the gravity test with the quality or performance of a product. The limits laid down qualitatively were nowadays so wide that accuracy meant little. The test had, however, a considerable significance from the quantitative point of view. When one talked about the gravity of a petroleum liquid from the quantitative point of view, one associated the term with the need for converting volumes into weights, where frequently a fairly high degree of accuracy was demanded but was not easy to attain. The bottle or pyknometer method yielded results of a relatively high order of accuracy; was there, then, any point in introducing further refinements, as one speaker had suggested, when, taking into account all the other errors which might be introduced in the measurement of a bulk quantity of an oil, all that could be hoped for was an accuracy of the order of one part in one or two thousand? Refinements in the specific-gravity method which resulted in the improvement of the "reproducibility" or "repeatability" figures from 0·0005 to 0·0002 or 0·0001 were, therefore, meaningless from a practical aspect. It was the intention of Sub-Committee No. 1 to see that what was set down in Standard Method 59/44 for qualitative work should be usable in part in another publication, for which they hoped to be responsible, on the whole question of measurement of oil quantitatively. He was certainly satisfied that the specific-gravity method as it appeared in the book to-day was, from the point of accuracy, and for qualitative purposes, adequate.

Mr. Beecroft supported the suggestion for including in the standard book an introduction on the significance of the tests.

Mr. Rosenfeld stressed the considerable interest of motor-vehicle operators in the properties of used crankcase oils, in view of existing limitations of supplies, and also for economical reasons. He, therefore, suggested that the Lubricants Sub-

Committee might consider the possibility of including, in a future edition of the "Standard Methods," suitable methods of testing used lubricating oils.

MR. E. A. EVANS said that Mr. Rosenfeld was particularly concerned with the oil-cleaning research which was going on at the Automobile Research Laboratory. He was naturally anxious to obtain some sort of uniformity in the method of expression of impurities in used oils. Clearly the term "carbon" had no particular meaning. The impurities in the used oil should be more definitely defined. Mr. Rosenfeld was seriously disturbed at the lack of uniformity, particularly when he was trying to describe things in his report which would be intelligible to a great number of people.

DR. BROOM said that perhaps this last point might engage the activities of the Nomenclature Sub-Committee. It would be remembered that there was a suggestion that that Sub-Committee should cover all the terms which appeared in the "Standard Methods," which might well mean that they ought to reconsider their terms of reference in the future to take this matter into account.

In listening to the general discussion that evening, he could not help thinking back on some of his reactions when he joined the Industry in 1930. He formed the opinion, rightly or wrongly, at that time that as far as lubrication was concerned, there was a great deal more art than science in it, and he did not know that he had changed that opinion so very much at the present time. Possibly they, as producers and marketers, viewed this matter differently from the user, who wanted to check up the material, but presumably in most cases it would be the material which had been proved satisfactory in service, and therefore the user, he imagined, was mainly concerned with seeing that he continued to get substantially identical material. As to the actual properties of additives and things of that nature, and their composition and so on, he rather agreed with a previous speaker that it was not so much the additive itself which was in question. The additives were put in to confer certain properties on certain oils, and provided they fulfilled this purpose, that should satisfy the user. He stood rather aghast at the chemical and technical ability which would be required even to identify some of these things, let alone estimate them.

DR. A. E. DUNSTAN said that he had the pleasant duty of asking the Meeting to convey its hearty appreciation of a monumental piece of work. He did not know whether the number of members of the Sub-Committees was as large as it was last year, when it stood at 146, which was getting on towards 10 per cent. of the total personnel of the Institute, but he could not think of any other body of technical and scientific men in which such a vast amount of good voluntary work was put into an effort of this kind.

He wanted to associate particularly with this vote of thanks the President, who was fortunately able to double with that high office the Chairmanship of the main Standardization Committee, and with him the Chairmen of the Sub-Committees and Panels, and his own three colleagues on the Editorial Committee. The real criterion of a good Chairman was said to be that he should know nothing at all about the subject, but be able to drive and induce his colleagues to do the work. That did not apply to Professor Garner for, in so far as knowing nothing of the subject was concerned, there were very few Chairmen who had his grip and grasp. But speaking of himself as Chairman of the Editorial Committee, the definition he had just given was a very good one, and therefore he wished particularly to include his colleagues on the Editorial Committee in a vote of thanks which should be extended to the President, the Chairmen of various Sub-Committees, and the Chairmen of the Panels, for what they had accomplished in the production of this very valuable publication.

The vote of thanks was put to the Meeting by Dr. Dunstan and carried with acclamation, and the Meeting terminated.

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